Remarks

Claims 1-93 are pending in this application. Claims 61, 67-69, 90, 92 and 93 have been withdrawn from consideration. Claims 1-60, 62-66, 70-89 and 91 are rejected.

Claim Objections

Claims 3, 8, 14, 17, 36, and 57 have been objected to by the Examiner. Applicants have amended claims 3, 8, and 14 to correct the typographical error, and claim 17 to correct the antecedent basis.

Regarding claim 36, Applicants respectfully point out that polymers of polyether-block-amides and acrylic esters of (meth)acrylic acid can be elastomeric. For reference, Applicants have attached references that describe one of each: a polyether-block amide (tradename PEBAX) and methyl acrylate used as elastomers.

Claim 57 has been cancelled. Applicants respectfully submit that all objections have been overcome.

Rejections under 35 USC 112

Claims 1-6, 11, 13-49, 51-58, 62-66, and 73-75 are rejected under 35 USC 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. Regarding claims 1, 19, 22-24, 27, 30-32, 62, 66 and 75, Applicants have amended the claims to delete the term "substantially." Applicants have also amended claims 1, 2, 11, 13, 51-56, and 73-74 to amend errors and clarify the invention.

Regarding claim 36, Applicants have amended the claim to correct the typographical error in placement of parentheticals. Regarding amended claim 36, Applicants respectfully disagree that a "(C1-C20) acrylic ester of (meth)acrylic acid" is not a compound. A simple search of the patent database reveals over 7500 hits on "acrylic ester" alone. The addition of alkyl proposed by the Examiner modifies "acrylic" and adds a limitation on the (C1-C20) carbon chain that Applicants did not delineate. It is readily apparent to one skilled in the art what compounds are identified, and Applicants maintain that no further amendment is necessary. Applicants have included a background on "Basic Acrylic Esters" as published by the Basic Acrylic

Monomers Manufacturers (BAMM) trade association, available on www.bamm.net. Claim 57 has been deleted.

With respect or claims 46 and 47, Applicants respectfully disagree. A composition can be for example a solid, and a solid can have a structural form such as a film or foam. A composition may also be in a liquid form; and a liquid can form a film. There is no structural limitation or lack thereof imputed by the term "composition." Applicants respectfully submit that all objections have been overcome.

Rejection under 35 USC 102

Claims 1, 3, 7, 8, 13-15, 62, 70, 75-77, 84 and 88 are rejected under 35 USC 102(b) as being anticipated by Bao et al. (CN 1308102, abstract). The Examiner has relied on the Abstract to disclose "a process of preparing a nanometer-sized (80-100nm) silver oxide" in which "before the solution is washed and vacuum dried to obtain the silver oxide, the ingredients in solution read on the presently claimed composition." Applicants note that an Abstract has been relied upon, and attach for the Examiner's reference an English translation relied on by the Applicants in responding to the Examiner's 102 and 103 rejections.

Applicants have amended claims 1, 7, 13, 62, 70, 75, 76, and 84 to specify the amount of water present in the composition. Because the solution in Bao discloses water in excess of 20 wt%, Bao cannot anticipate the claims as amended. Applicants respectfully submit that the rejection has been overcome.

Rejection under 35 USC 103

Claims 85-87 are rejected under 35 USC 103(a) as being obvious over Boa et al. (CN 1308102, abstract). The Examiner notes that Bao fails to disclose the mixing order. Applicants respectfully traverse the rejection.

The Examiner bears the initial burden of factually supporting any *prima facie* conclusion of obviousness. In re Fine, 5 U.S.P.Q.2d 1596, 1598 (Fed. Cir. 1988). To establish a *prima facie* case of obviousness, three criteria must be met. First, the prior art reference (or references) must teach or suggest all of the claim limitations. Second, there must be some suggestion or motivation, either in the cited reference (or references), or in the knowledge generally available to one of ordinary skill in the art,

to modify the reference or combine reference teachings. Third, there must be a reasonable expectation of success. M.P.E.P. § 2142 (citations omitted).

Applicants submit that Bao fails to teach or suggest a polymer composition with water present in an amount of 1 to 20 wt% based on the total weight of the polymer composition or dispersing the metal oxide within the hydrophilic polymer as claimed in amended claim 84 (from which claim 85-87 depend). Thus, Bao fails to teach all the claim limitations of claims 85-87.

Further, the Examiner fails to identify disclosure in Bao to support the motivation to modify the solution of Bao. The teaching or suggestion to make the modification of a single reference and the reasonable expectation of success must both be found in the prior art, and not based on Applicants' disclosure. See M.P.E.P §706.02(j).

Claims 1-4, 6-9, 11, 12, 19-39, 42-45, 48, 49, 50, 52, 53, 55-60, 75 and 89 are rejected under 35 USC 103(a) as being obvious over Asmus (US 5,270,358) alone or in view of Laurin et al. (US 4,603,152). The Examiner cites Asmus to disclose a composite "comprising 1-95 wt% a gel having a size of 1-600 microns containing hydrocolloid (i.e., a hydrophilic polymer) and a swelling agent; antimicrobial agents such as silver oxide; pressure sensitive adhesive (i.e., matrix polymer); water; and other additives."(citations omitted).

Applicants respectfully traverse the rejection for several reasons. First, the Examiner states that 7-9, 11-12, 19-23, 26-31, 34-39, 42-45, 52-53, and 55-59 are product-by-process claims, and Asmus discloses the use of silver oxide, thus the final composition of Asmus must be the same as the composition claimed by Applicants. However, the Examiner acknowledges that Asmus fails to disclose the particle size of the silver oxide.

As Applicants describe in the Specification beginning with the paragraph on page 12, line 6:

> Substantially all of the dispersed silver, zinc, and copper compounds have an average particle size less than 1 micron in size. <u>By utilizing a process that solubilizes the metal compound</u>, either through use of a soluble metal compound that is converted in-situ to the corresponding metal oxide with a hydroxide source, or by complexing the metal oxide using an ammonia source in situ, <u>the resulting dispersed metal oxides form particles within the hydrophilic polymer.</u> Average particles sizes less than 1 micron are provided in part by the tendency of the metal oxide to form a complex with the hydrophilic polymer. The small particle size allows accelerated dissolution based on the high surface area to mass ratio of the particle. [emphasis added]

Thus, contrary to the Examiner's assertion, the process of making the product does effect the composition as claimed by Applicants, such as by dispersing the metal oxides within the hydrophilic polymer. Asmus makes no disclosure regarding the silver oxide particle size, or whether it is dispersed within the hydrocolloid of Asmus. Applicants' disclosure cannot be used in hindsight to provide the motivation to modify Asmus. Further, Asmsu provides no disclosure of adding a hydroxide source to accomplish the dispersion of the silver oxide. Even if silver oxide particles (i.e., less than 1 micron) were mixed as an additive in the composition of Asmus (which Applicants maintain that Asmus does not teach or suggest), the mixture would not necessarily contain silver oxide dispersed within the hydrophilic polymer.

The Examiner further concludes that because the swelled gel of Asmus is between 1 and 600 microns, then "the unswelled state of the gel particles have a particle size less than 1 micron." Applicants respectfully disagree. Rather, Asmus discloses in the Example 1 that the majority of particles used in Asmus are between 25 and 200 microns. (col. 21, lines 24-26).

The Examiner further states that because the microbial agents are like thos of Applicants' claims, the microbial agents "intrinsically have a solubility in water of at least 0.1 gram per liter in water." Applicants disagree and note that silver oxide has a solubility less than 0.1 gram per liter in water. (Handbook of Chemistry and Physics, 64^{th} Ed. 1983, p. B-137). Rather, it is the presence of the hydroxide source that converts a silver compound (having a solubility in water of at least 0.1 gram per liter) to the corresponding silver oxide (with a solubility less than 0.1 gram per liter in water).

The Examiner further cites Laurin for the teaching of the "submicron antimicrobial agent." Laurin does not cure the deficiencies of the Asmus reference discussed above.

Claims 5, 10, 40 and 41 are additionally rejected under 35 USC 103(a) as being obvious over Asmus (US 5,270,358) alone or in view of Laurin et al. (US 4,603,152) and further in view of Ahmed et al. (US 6,458,877). Ahmed is cited to teach the use of a quaternary ammonium salt of an organic polymer. The addition of Ahmed as a reference does not cure the deficiencies of Asmus discussed above.

Claims 13-18, 84-87 and 92 are additionally rejected under 35 USC 103(a) as being obvious over Asmus (US 5,270,358) alone or in view of Laurin et al. (US 4,603,152) and further in view of Yan et al. (US 2003/0185889). The Examiner cites Yan for the disclosure of ammonia to increase the solubility of silver oxide in water. Yan teaches a colloidal nanosilver solution. The addition of Yan as a reference does not cure the deficiencies of Asmus discussed above.

Claims 62, 64-66, 70, 72-74, 76-78 and 80-83 are rejected under 35 USC 103(a) as being obvious over Asmus (US 5,270,358) alone or in view of Laurin et al. (US 4,603,152) and further in view of Boa et al. (CN 1308102, abstract). The Examiner cites Bao for the disclosure of the hydroxide source. As discussed above, Bao teaches a nanosilver oxide solution. The addition of Bao as a reference does not cure the deficiencies of Asmus discussed above.

Claim 79 is rejected under 35 USC 103(a) as being obvious over Asmus (US 5,270,358) alone or in view of Laurin et al. (US 4,603,152) and further in view of Boa et al. (CN 1308102, abstract) and Ahmed et al. (US 6,458,877). Ahmed is cited to teach the use of a quaternary ammonium salt of an organic polymer. The addition of Ahmed as a reference does not cure the deficiencies of Asmus discussed above.

Claims 63 and 71 are rejected under 35 USC 103(a) as being obvious over Asmus (US 5,270,358) alone or in view of Laurin et al. (US 4,603,152) and further in view of Bao et al. (CN 1308102, abstract) and Antelman (US 6,436,420). The Examiner cites Antelman for the disclosure of higher valence silver compounds. The addition of Antelman as a reference does not cure the deficiencies of Asmus discussed above.

Double Patenting

The Examiner provisionally rejects claims 1-12, 19-60, 75 and 89 under the doctrine of obviousness-type double patenting over claims 1-15, 19-21, 27-39, 41-46, 49, 51, 53-63 and 70 of co-assigned Application No. 10/728,577 (published as US 2004/0180093) alone or in view of Laurin et al. (US 4,603,152). Applicants respectfully submit that the above patents are commonly owned with this application. Upon the identification of otherwise allowable subject matter, Applicants reserve their right to argue the patentable distinctness of the claims over co-assigned Application No. 10/728,577 (published as US 2004/0180093) alone or in view of Laurin, or

alternatively offer a terminal disclaimer to cure any remaining obviousness-type double patenting rejections. As a preliminary matter, applicant submits that the Examiner has failed to establish a *prima facie* conclusion of obviousness over co-assigned Application No. 10/728,577 for the same reasons as given to traverse the obviousness rejection over Asmus.

The Examiner further states that claims 1-12, 19-60, 75 and 89 are directed to an invention not patentably distinct from claims 1-15, 19-21, 27-39, 41-46, 49, 51, 53-63 and 70 of commonly assigned copending Application No. 10/728,577 (published as US 2004/0180093). Applicants request further clarification on the basis of this rejection as it is not clear whether this is an amplification of the obviousness-type double patenting rejection, a 102(e) rejection or a 103(a) rejection, or some other statutory rejection.

Conclusion

All outstanding objections and rejections are believed to have been met and overcome. If a telephonic conference with Applicants' undersigned representative would be useful in advancing the prosecution of the present application, the Examiner is invited to contact the undersigned at (651) 733-2180. A notice of allowance for all pending claims is respectfully solicited.

Respectfully submitted,

Nancy M. Lambert

Nancy M. Lambert
Registration No. 44,856
Attorney for Applicants

NML:jlh/#75943 - 59405US002 Resp to OA 11-03-05
Office of Intellectual Property Counsel
3M Innovative Properties Company
P.O. Box 33427
St. Paul, Minnesota 55133-3427
(651) 733-2180
Facsimile: (651) 736-3833

Dated: May 3, 2006

Pebax®

Advance

Home > Products & Markets > Pebax®

- Product info
- Literature
- News
- Tradeshows
- Contact

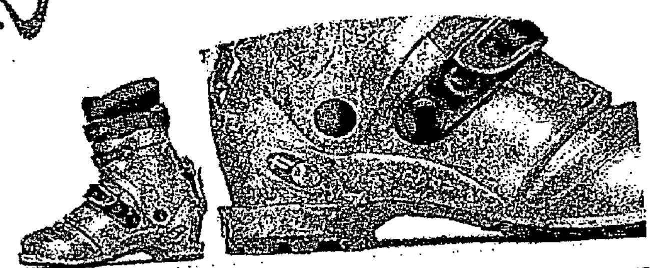
More Arkema websites

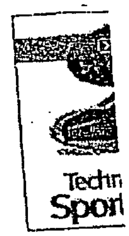
Select a website

MSDS/SDS
Technical Data

PRODUCT FINDER

pebax





Arkem

- Electi Electi
- Cons
- Sport
- Packa
- Hygie Beaut
- Plasti

Pebax® polyether block amides are plasticizer-free thermoplastic elastomers belonging to the engineering polymer family. They are easy to process by injection molding and profile or film extrusion. Pebax® can be easily melt blended with other polymers.

Their unique chemistry (polyether block amide - PEBA) allows Pebax® to achieve a wide range of physical and mechanical properties by varying the monomeric block types and ratios.

The Pebax® product range extends from grades with properties of polyamide to grades with properties more like elastomers.

The remarkable processing performance of Pebax® makes them an ideal material for components requiring:

- Light weight;
- Great flexibility (extensive range);
- Resiliency;
- Very good dynamic properties due to low hysteresis (alternative flexure);
- High strength;
- Outstanding properties at low temperature (impact resistance, low rigification);
- Easy processing;
- Good resistance to most chemicals;
- Ability to be impregnated with fragrances;
- Outstanding impact resistance at low temperatures

PRODUCTS > INDUSTRIAL CHEMICALS > FUCTIONAL MONOMER >

METHYL METHACRYLATE

PRODUCT IDENTIFICATION	
CAS NO.	80-62-6
EINECS NO.	201-297-1
FORMULA	$H_2C=C(CH_3)COOCH_3$
MOL WT.	100.12
H.S. CODE	2916.14
TOXICITY	Oral rat LD50: 7872 mg/kg
SYNONYMS	MMA; 2-Methyl 2-propenoic acid, methyl ester; Methacrylic acid, methyl este

2-(Methoxycarbonyl)-1-propene; 2-Methyl propenoic acid, methyl ester; 2-Propenoic acid, 2-methyl-, methyl est
methyl-2-propenoate 2-Methyl-2-propenoic acid methyl ester; Methacrylate De Methyle (French);
acid, 2-methyl-, methyl ester; Metakrylan metylu (Polish); Methyl 2-methyl Propenoate; Methyl 2-methyl-2-propenoate;
Methacrylsaeuremethyl Ester (German); Methyl 2-methyl Propenoate; Methylester Kyseliny Methakrylove; Methylmeth·
apha-methyl Acrylate; Methyl-methacrylat (German); Methylester Kyseliny Methakrylove; Methylpropylene-2-carboxylate;
(Dutch); Methylmethacrylate; Metil Metacrilato (Italian); MME; Methylpropylene-2-carboxylate;

DERIVATION	esterification of methacrylamide sulfate with methanol
CLASSIFICATION	
PHYSICAL AND CHEMICAL PROPERTIES	
PHYSICAL STATE	clear liquid
MELTING POINT	-48 C
BOILING POINT	101 C
SPECIFIC GRAVITY	0.942 - 0.946
SOLUBILITY IN WATER	Insoluble
pH	
VAPOR DENSITY	3.6
AUTOIGNITION	435 C
NFPA RATINGS	Health: 2; Flammability: 3; Reactivity: 2
REFRACTIVE INDEX	1.4118 - 1.4125
FLASH POINT	11 C
STABILITY	The stability depends upon dissolved oxygen and MEHQ inhibitor. The presei oxygen is necessary for inhibitor to prevent polymerization.

DESCRIPTION & APPLICATIONS

Acrylic acid is the simplest unsaturated carboxylic acid which has double bond and carboxyl group ir molecule with the formula CH_2=CHCOOH. The vinyl group is attached to the carbonyl carbon directly systemic name is 2-propenoic acid. Acrylic acid has two reaction points or functional groups required polymerization process. Purified (glacial) acrylic acid is a clear, colorless liquid with a characteristic ac miscible with water, alcohols and ethers. Acrylic acid is produced from C 3 refinery. Acrylic acid undei typical reactions of a carboxylic acid and forms acrylic esters - basic alkyl esters are methyl, butyl, eth and 2-ethylhexyl acrylate. Acrylic acid and its esters undergo the reactions of the double bond which combine with themselves or other monomers (e.g amides, methacrylates, acrylonitrile, vinyl, styrene ai butadiene) to form homopolymers or co-polymers which are used in the production of coatings, adhe elastomers super absorbent polymers, flocculants, as well as fibres and plastics. Acrylate polymers sho range of properties dependent on the type of the monomers and reaction conditions.

NOTE

Alkyl acrylates are clear, volatile liquid; slightly soluble in water and complete soluble in alcohols, ethe
almost organic solvents; Acrylate esters containing a double bond and functional carboxyl group are
chiefly as a monomer or co-monomer in making acrylic and modacrylic fibres. It is used in formulating
dispersions for paints, inks, and adhesives. It is used in making cleaning products, antioxidant agents, c
surfactants. It is used in making aqueous resins and dispersions for textiles and papers. Methyl acrylate
making vitamin B1.

	Methyl acrylate	Ethyl acrylate	Butyl acrylate	2-Ethylhex
CAS RN	96-33-3	140-88-5	141-32-2	103-
Formula (MW)	CH_2=$CHCOOCH_3$	CH_2=$CHCOOCH_2CH_3$	CH_2=$CHCOO(CH_2)_3CH_3$	CH_2=CHC
Mole Weight	86.09	100.12	128.17	18
Specific Gravity	0.9567	0.923	0.9015	0.8
Viscosity cP (20 C)	0.49	0.6	0.9	1
Boiling Point C at 760 mm Hg	79.6	100	147	21 (Decor
Melting Point C	< -75	-72	-64	-
Solubility in water	slightly soluble 30 - 60 (g/l)	slightly soluble (15 g/l)	Slightly soluble (2-7 g/l)	negl

Methyl methacrylate (MMA) is ester of the unsaturated C4 carboxylic acid. The term of metha indicat
additional methyl group attached to the alpha carbon of acrylic acid. Methyl methacrylate is a flamr
colorless liquid; melting at -48 C, boiling at 101 C, soluble in the most organic solvents but insoluble in w
prepared by the esterification of methacrylamide sulfate with methanol. (The reaction of acetone an
cyanide forms acetone cyanohydrin, which is further treated with sulfuric acid to produce methacryla
sulfate). Ammonium bisulfate is a byproduct. MMA is produced commercially also from C4 route (isob
and tert-butyl alcohol) through two oxidation process. This process don't need sulphuric acid and no
products. MMA is the monomer to make polymethyl methacrylate (PMMA) used as a shatterproof rep
for glass. It is a key ingredient in the production of cast and extruded acrylic sheet, acrylic emulsions, n
powders and extrusion resins. Polymers and copolymers of methyl methacrylate are also used in undis
surface coatings, adhesives, sealants, impact modifiers, emulsion polymers, surgical bone cements, pc
applications, vinyl siding and other construction materials.

Acrylics including (meth)acrylic acids, acrylic esters, and acrylic compounds containing reactive halo
and amide groups are versatile monomers forming any class of hard, soft, resilient and transparent syn
plastics or resins and viscous oils by varying the starting materials and the polymerization processes. Th
monomers can be either homopolymerized or be copolymerized with other type monomers capable
polymerized. The resultant homopolymers can provide abundance hydrophilic property groups. Copc
be either hydrophilic or hydrophobic. Sodium acrylate is copolymerized with acrylamide to make an c
copolymer used as a flocculant in water treatment. Acrylic esters copolymers with minor amounts of c
functional monomer containing a reactive halogen or ethylenically unsaturated ester can form inter-li
polymer chains that display good heat and oil resistance. Homopolymers and copolymers have a vari
industrial applications including;

- Plastics
- Textiles
- Thickening Agents
- Dispersing Agents
- Surfactants
- Chelating Agents
- Adhesives

- Water-based coatings
- Water Treatment

SALES SPECIFICATION	
APPEARANCE	clear liquid
PURITY	99.8% min
COLOR, APHA	10 max
FREE ACID	0.005% max
SPECIFIC GRAVITY	0.942 - 0.946
WATER	0.05% max
INHIBITOR	5 ±1 ppm (Monomethyl Ether Hydroquinone)
TRANSPORTATION	
PACKING	180kgs in drum
HAZARD CLASS	3 (Packing Group: II)
UN NO.	1247
REMARKS	

We are pleased to receive your any questions or remarks. You may contact us at the following number address for additional information.
Fax : + 82 + 2 + 783-8063 , + 82 + 2 + 3775-3073 (Seoul, Korea)

HOME

BASIC ACRYLIC MONOMER MANUFACTURERS, INC.

Basic Acrylic Esters:

Background Information

ABOUT BAMM

The Basic Acrylic Monomer Manufacturers, Inc. (BAMM) is a trade association of manufacturers and importers of acrylic acid and its basic esters including butyl acrylate, ethyl acrylate, methyl acrylate and 2-ethylhexyl acrylate. BAMM current membership includes: BASF Corporation; ATOFINA Chemicals, Inc.; Celanese Ltd; Rohm and Haas Company; and The Dow Chemical Company.

ABOUT THIS SUMMARY

Basic Acrylic Monomers - acrylic acid and its esters - are versatile chemicals that have widespread use. The manufacturers, and others, have conducted scientific studies on the health and environmental effects of these products. This brochure provides a summary of technical, exposure, and regulatory information for use by government officials, health professionals, and others who would like a brief introduction to the data. This brochure includes data that BAMM is aware of as of the date of publication. BAMM has been careful to accurately summarize the data and conclusions; however, newer data may be or become available.

Users should also consult their suppliers' Material Safety Data Sheet (MSDS) for up-to-date information and precautions for safe handling and use of these chemical products.

INTRODUCTION

Acrylic acid has served, for more than 30 years, as an essential building block in the production of some of our most commonly used industrial and consumer products.

Approximately two-thirds of the acrylic acid manufactured in the United States is used to produce acrylic esters - methyl acrylate, butyl acrylate, ethyl acrylate, and 2-ethylhexyl acrylate - which, when polymerized (see below), are ingredients in paints, coatings, textiles, adhesives, plastics, and many other applications.

The remaining one-third of the acrylic acid is used to produce polyacrylic acid, or crosslinked polyacrylic acid compounds, which have been successfully used in the manufacture of hygienic products, detergents, and waste water treatment chemicals.

Polymerization

Acrylic monomers are highly reactive chemicals and, therefore, are used nearly exclusively as intermediates in the production of other materials. For example, individual molecules of acrylic acid or esters, called "monomers," readily combine with themselves or other monomers to form long chains of repeating units, or "polymers." The polymers have different physical and chemical properties than the constituent monomers.

These polymers can be used in the production of goods that for decades have provided added benefits and convenience to consumers and manufacturers worldwide.

PHYSICAL PROPERTIES

	Methyl	Ethyl	Butyl	2-Ethylhexyl

Basic Acrylic Esters:

	Acrylate	Acrylate	Acrylate	Acrylate
Molecular Formula	$C_4H_6O_2$	$C_5H_8O_2$	$C_7H_{12}O_2$	$C_{11}H_{20}O_2$
Molecular Weight	86	100	128	184
CAS Number	96-33-3	140-88-5	141-32-2	103-11-7
Boiling Point (°C)	80	100	148	216
Freezing Point (°C)	<-76	<-72	<-65	<-76
Density (20/20 °C)	0.957	0.923	0.900	0.886
Flash Point (Tag Closed Cup °C)	-3	8	41	87

EXPOSURE TO ACRYLIC ESTERS

Acrylate vapor has a very strong acrid odor that leads to early detection of any potential releases. The following table contains a list of current standards for levels to which the standard-setting bodies consider that most workers can be exposed during an 8-hour workday without harmful effects, as well as the results of tests used to determine the levels at which the acrylates usually will be smelled. As illustrated, acrylate odor usually will be detected before it reaches the levels of the current standards. While unpleasant, smelling acrylate vapor is not necessarily indicative that such levels have been exceeded.

	Methyl Acrylate	Ethyl Acrylate	Butyl Acrylate	2-Ethylhexyl Acrylate
OSHA Permissible Exposure Limit (ppm)	10, skin	25, skin	None	None
ACGIH Threshold Limit Value® (ppm)	2, skin, sensitizer, A4	5, A4 15 ppm STEL	2, A4, sensitizer	Not established
Odor Threshold (ppm)	0.014 to 0.020	0.00036 to 0.001	0.0001 to 0.009	0.016 to 0.18

Because consumer products contain only trace levels of acrylic esters as a result of the polymerization process, consumers are not generally exposed to these compounds in finished products. Although potential for exposure does exist during acrylic ester manufacture, transportation, and use, enclosed systems limit the exposure to worker populations and nearby communities. Employees and contractors involved in acrylic ester manufacturing should wear appropriate safety equipment and undergo special training.

In spite of the manufacturer's efforts to contain the manufacturing process, vapors can potentially escape from leaks in the piping system, during repair or replacement of the piping system (including tanks and reactors), or during removal of samples for quality control purposes. Worker exposure should be monitored in manufacturing facilities with specialized monitoring systems. Because odors will be detected at such low levels, leaks should be detected and repaired before a product can cause adverse health effects

Exposure could also occur during loading, unloading, and transportation of tank trucks, railroad tankers, barges, and drums. However, dedicated systems designed to handle acrylic esters are typically used for loading and unloading purposes and procedures should be in place to prevent spills or leaks during transportation.

TOXICOLOGY PROFILE

Basic Acrylic Esters:

The likely primary routes of human exposure to the acrylic esters are skin contact and inhalation; toxicity is due to tissue damage at the site of contact. The undiluted esters are moderately irritating to corrosive liquids that can cause permanent tissue damage upon direct contact with the body. Contact with dilute solutions of the esters or monomer vapors can be irritating to the skin, eyes and respiratory tract.

In animal studies, the irritation caused by repeated exposure to acrylic esters vapor at levels exceeding the TLV® has resulted in nasal lesions. The esters have caused skin sensitization, and individuals allergic to one ester may exhibit a sensitization reaction when exposed to other esters.

In general, the esters that are absorbed are rapidly broken down in the body by two different mechanisms. The resulting metabolites are eliminated via expired air and urine.

Toxicity associated with repeated exposures of experimental animals to acrylate esters typically has been comprised of changes in body and organ weights as well as clinical chemistry. Although the National Toxicology Program has determined these results are not relevant to man, ethyl acrylate has caused tumors in animals when large doses were injected by tubes directly into the animals' forestomachs. Except for dermal and nasal lesions which have been observed at the site of contact, the microscopic examination of other body tissues post-exposure has indicated no significant difference compared to non-exposed animals.

The acute toxicity of acrylic acid to fish and invertebrates ranged from "slightly" toxic to "practically non-toxic." The acute toxicity of the acrylic esters was "moderately" toxic. Acrylic acid and the acrylic esters tested do not accumulate in the environment. If released to surface water, acrylic acid and the esters would all be rapidly biodegraded; some portion should volatilize to the air.

REGULATORY STANDARDS

The manufacture, transportation, and use of basic acrylates are regulated or addressed by a number of government agencies and other expert groups to control exposure to workers and the environment. The following table records major U.S. federal regulatory and similar information as of June 1999. Such regulations frequently change, and many states and localities adopt their own regulations. Therefore, you should consult applicable laws and regulations, as well as the manufacturer's MSDS, for current requirements.

REGULATORY STANDARDS

	Methyl Acrylate	Ethyl Acrylate	Butyl Acrylate	2-Ethylhexyl Acrylate
OSHA PEL	10 ppm, skin	25 ppm, skin	None	None
ACGIH TLV®	2 ppm, skin, sensitizer	5 ppm 15 ppm STEL	2 ppm, sensitizer	None
IARC	3	2B (Suspect Human Carcinogen)	3	None
NTP BRC	no	no	no	no
DOT Hazard Class	flammable	flammable, corrosive	flammable	no
SARA/CERCLA RQ	no	1000 lbs	no	no
SARA 313	yes	yes	yes	no
RCRA	no	yes	no	no

Basic Acrylic Esters:

CAA	no	yes	no	no
CWA	no	no	no	no
FDA Uses[1]	yes	yes	yes	yes
NFPA Rating: [2]				
· Health	2	2	2	2
· Flammability	3	3	2	2
· Reactivity	2	2	2	2

[1] *Acrylic esters used as monomer to make specific polymers are listed for certain, specific indirect food additive uses.*

[2] *NFPA Rating Definitions: 2/Health–Materials hazardous to health, but areas may be entered with full-face mask, self-contained breathing apparatus which provides eye protection; 2/Flammability–Materials that must be moderately heated before ignition will occur; 3/Flammability–Materials that can be ignited under almost all normal temperature conditions; 2/Reactivity–Materials that (in themselves) are normally unstable and readily undergo violent chemical change but do not detonate.*

[3] *Delisted from the NTP Report on Carcinogens in May 2000*

ACRONYM LISTING

ACGIH TLV®	American Conference of Governmental Industrial Hygienists, Threshold Limit Value®
CAA	Clean Air Act, §112(b)
CWA	Clean Water Act, §§116.4, 112 App. D, 131.36, 401.15, 423 App. A.
DOT Hazard Class	Dept. Of Transportation: Hazard Class 49 C.F.R. §172.101
FDA	Food & Drug Administration: 21 C.F.R. Parts 174 to 178
IARC	International Agency for Research on Cancer
NFPA	National Fire Protection Association
NTP RC	National Toxicology Program Report on Carcinogens
OSHA PEL	Occupational Safety and Health Administration, Permissible Exposure Limit, 29 C.F.R., Table Z, §1910.1200
RCRA	RCRA - Resource Conservation and Recovery Act, 40 C.F.R. §261.33
SARA/CERCLA RQ	Superfund Amendments and Reauthorization Act (1986), 40 C.F.R. §375/Comprehensive Environmental Response, Compensation, & Liability Act (1980), 40 C.F.R. §302.4 Reportable Quantity
SARA 313	Section 313 of Title III of SARA - Toxic Release Inventory Reporting & Community Right-To-Know, 40 C.F.R. §72.65

FOR MORE INFORMATION

For more information, please contact your supplier or BAMM at the following address:

941 Rhonda Place SE
Leesburg, VA 20175
Office: (703) 669-5688
Fax: (703) 669-5689
Email: ehunt@adelphia.net

NOTICE

59405US002

[19] Intellectual Property Bureau of the Peoples' Republic of China

[51] Int. Cl[7]
C09C 3/00

[12] Novel Application Patent Public Announcement

[21] Application No.0118083.3

[43] Announcement Date: October 6, 2004 [11] Announcement No. CN 1169883C

[21] Application No.: 01108083.3
[22] Date of Application: February 7, 2001

[71] Assignee: University of Nanjing
Address: No. 22, Han Kou Road
Nanjing, Jiangsu Province, People's Republic of China 210093
[72] Inventors: ZHOU Quan-fa, XU Zheng, and BAO Jian-chun
Examiner: YIN Chao-hui

[74] Patent Representative Agency: Nanjing Zhi Shi (Knowledge) Law Office
Representative: Huang Jia-dong

Patent Claims 1 page; Descriptions 3 pages

[54] Name of Invention: Nanometer Grade Silver Oxide and the Process for Its Manufacturing

[57] Summary

A nanometer grade silver oxide; the average particle size of which is 80 – 100 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm. The process of preparation is to dissolve silver nitrate in water, gradually adding concentrated ammonia water while stirring, to obtain an ammoniacal silver solution. Solid sodium hydroxide and a protective agent are then dissolved separately in water while stirring, to obtain an alkali solution. The protective agent may be polyvinyl-pyrrolidone or polyvinyl alcohol. The ammoniacal silver solution is gradually added to the alkali solution while stirring; after further through stirring, the solution is centrifuged to separate the silver oxide precipitate, which is then washed and vacuum dried; obtaining a nanometer grade silver oxide, with good stability and dispersibility, as well as uniformly distributed particle size. The process may be used for industrial manufacturing.

Published by Intellectual Property Publisher
ISSN 1008 – 4274

Patent Claims

1. A nanometer grade silver oxide, the characteristics of which are that the average particle size is 80 – 100 mm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

2. A process for manufacturing the nanometer grade silver oxide as mentioned in Claim 1, the characteristic of which are that an aqueous solution of silver nitrate is prepared, followed by the addition of ammonia water to obtain a ammoniacal silver solution, which is then gradually added to a sodium hydroxide solution with pre-added protective agent, forming super fine silver oxide precipitates; after filtration, washing, and drying, super fine silver oxide with an average particle size of 80 – 100 nm is obtained; the difference between the largest particle size and the smallest particle size is ≤ 5 nm; the protective agent mentioned is polyvinyl-pyrrolidone or polyvinyl alcohol.

Descriptions

Nanometer Grade Silver Oxide and the Process for Its Preparation

This invention is pertaining to nanometer grade silver oxide and the process for its preparation.

Silver oxide (Ag_2O) has been widely used in the industry for electronic components, button batteries, preservatives or antiseptics, cleaning agents, glass coloring agent and polishing agent, as well as chemical catalysts, etc.. Thirty percent of industrial silver usage is for production of silver oxide (or silver nitrate), in which the silver oxide consumption by the electronic component industry accounts for 90 % of the total silver oxide. Currently, domestic silver oxide are primarily prepared from the reaction of a silver nitrate solution with a sodium hydroxide solution to obtain a silver oxide precipitate, which is then washed, separated, and dried to obtain the silver oxide finish product. The major technical indexes (indicators) of the product include silver oxide content, clarity test and nitric acid insoluble materials, free alkali, nitrate salts, hydrochloric acid un-precipitated material content, and drying weight loss, etc..

The major existing issues include:

1. There is no particle size index in current product; the particle size is difficult to meet the requirements of various industries. The silver oxide used in the electronic component industry is usually prepared into a silver oxide paste, which requires extended ball milling levigation to reduce the size (require approximately 7 days) before mixing with other materials, which is then printed onto a device substrate (chip) through silk screening. The particle size of silver oxide is directly impacting the quality of the prepared silver paste and the cost of silk screen, which is in turn affecting the quality of the device. The requirement of the silver oxide particle size for the electronic industry is "the smaller the better". Current manufacturing technologies used both domestically and abroad are difficult to meet these requirements. Silver oxide used in the industries for chemical catalyst, medical antiseptics, glass coloring, etc., also requires a particle size of "the smaller the better".

2. Although domestically produced silver oxide is meeting the national purity standard, however, due to current processing technology, the particle size of current product is too large, making it easy to enclose impurities; it is very difficult to obtain high or super high purity grade silver oxide.

The objective of this invention is to provide a nanometer grade silver oxide, with good stability and dispersibility, and a uniformly distributed particle size, as well as a process for industrial manufacturing.

Technical thinking behind this invention: When a silver nitrate solution is reacting with a sodium hydroxide solution, the Ag^+ ion concentration in the solution is usually too high, the rate of growth of the silver oxide particle produced in the reaction system is too fast; at the same time, it is very easy to form aggregates between the particles, as a result, the particle size of the silver oxide produced is too large. If the Ag^+ ion concentration can be reduced during reaction, allowing the formation of silver oxide to speed up while the growth rate to slow down; at the same time, if the silver oxide particles are protected once they are formed, to prevent aggregate from forming, then, a silver oxide product with very small particle size can be prepared.

Therefore, the technical schemes of this invention are as follows:

A nanometer grade silver oxide, with an average particle size of ≤100 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

A process for manufacturing the nanometer grade silver oxide, which involves the preparation of an aqueous solution of silver nitrate, concentrated ammonia water is added to obtain an ammoniacal silver solution, which is then gradually added to a sodium hydroxide solution with pre-added protective agent, forming super fine silver oxide precipitates; after filtration, washing, and drying, super fine silver oxide with an average particle size of 80 – 100 nm is obtained;

the protective agent mentioned above may be polyvinyl-pyrrolidone or polyvinyl alcohol.

The specific process involves:

Dissolve 50 – 100 (parts by weight, same hereafter) of silver nitrate in 300 parts of water, add 100 – 400 parts of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 parts of water is added to 20 – 40 parts of solid sodium hydroxide and 0.2 – 1.0 parts of protective agent; it is stirred until dissolved to obtain an alkali solution. The protective agent may either be polyvinyl-pyrrolidone or polyvinyl alcohol. The ammoniacal silver solution mentioned above is gradually added to the alkali solution mentioned above while stirring. After the solution is further thoroughly stirred for 4 – 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared by the method of this invention has a particle size of 80 – 100 nm, has good stability and dispersibility, with a uniform particle size distribution.

Using the manufacturing process of this invention, the silver oxide obtained has an average particle size of less than 100 nm, the difference between the largest particle size and the smallest particle size is ≤ 5 nm; since a protective agent is being used, it has very good stability and dispersibility.

Application Example 1

Dissolve 50 Kg of silver nitrate in 300 L of water, add 100 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 40 Kg of solid sodium hydroxide and 0.2 Kg of protective agent (polyvinyl-pyrrolidone, or PVP, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 4 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 88 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 2

Dissolve 100 Kg of silver nitrate in 300 L of water, add 100 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water id added to 25 Kg of solid sodium hydroxide and 1.0 Kg of protective agent (polyvinyl-pyrrolidone, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 100 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 3

Dissolve 100 Kg of silver nitrate in 300 L of water, add 400 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 40 Kg of solid sodium hydroxide and 1.0 Kg of protective agent (polyvinyl-pyrrolidone, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 82 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 4

Dissolve 50 Kg of silver nitrate in 300 L of water, add 400 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 40 Kg of solid sodium hydroxide and 1.0 Kg of protective agent (polyvinyl-pyrrolidone, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 80 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 5

Dissolve 50 Kg of silver nitrate in 300 L of water, add 100 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 15 Kg of solid sodium hydroxide and 0.2 Kg of protective agent (polyvinyl-pyrrolidone, or PVP, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 92 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

PHYSICAL CONSTANTS OF INORGANIC COMPOUNDS (Continued)

No.	Name	Synonyms and Formulae	Mol. wt.	Crystalline form, properties and index of refraction	Density or spec. gravity	Melting point, °C	Boiling point, °C	Solubility, in grams per 100 cc		
								Cold water	Hot water	Other solvents
	Silver									
s168	nitrite	$AgNO_2$	153.88	wh, rhomb	4.453^{26}	d 140	...	0.155^{0}	1.363^{60}	s ac a, NH_4OH; i al
s169	nitroplatinite	$Ag_2[Pt(NO_2)_4]$	594.85	yel-brn monocl pr	...	d 100	...	sl s	s	...
s170	nitroprusside	$Ag_2[FeNO(CN)_5]$	431.68	lt pink	...	...	...	i	...	s NH_4OH; i al, HNO_3
s171	oxalate	$Ag_2C_2O_4$	303.76	col cr	5.029^{4}	expl 140	...	0.00339^{18}	..	s KCN, NH_4OH, a
s172	oxide	Ag_2O	231.74	br-blk, cub	$7.143^{16.6}$	d 230	...	0.0013^{20}	0.0053^{80}	s a, KCN, NH_4OH, al
s173	oxide, per	Ag_2O_2 (or AgO)	247.74	gray-blk, cub	7.44	d>100	...	i	...	s H_2SO_4, HNO_3, NH_4OH
s174	palmitate	$AgC_{16}H_{31}O_2$	363.29	wh, greasy powd	...	209	...	0.0012^{20}	0.006^{25}	0.007^{15} eth; 0.006^{25} al
s175	*meta*phosphate	$AgPO_3$	186.84	wh, amorph	6.37	ca 482	...	i	...	s HNO_3, NH_4OH
s176	*ortho*phosphate	Ag_3PO_4	418.58	yel, cub	6.370^{25}	849	...	$0.00065^{19.5}$	...	s a, KCN, NH_4OH, NH_3
s177	*ortho*phosphate, mono-H	Ag_2HPO_4	311.75	wh, trig	1.8036	d 110	...	...	...	...
s178	*pyro*phosphate	$Ag_4P_2O_7$	605.42	wh	$5.306^{7.5}$	585	...	i	i	s a, NH_4OH, KCN, ac a
s179	propionate	$AgC_3H_5O_2$	180.94	wh leaf or need	2.687^{25}_{4}	...	...	0.842^{20}	2.04^{80}	...
s180	perrhenate	$AgReO_4$	358.07	wh cr, tetrag or rhomb	7.05	430	...	0.32^{20}	...	...
s181	salicylate	$AgC_7H_5O_3$	244.99	wh to redsh-wh cr	...	...	...	sl s	...	s al
s182	selenate	Ag_2SeO_4	358.73	wh, orthorhomb cr	5.72	...	...	0.118^{20}	...	...
s183	selenide	Ag_2Se	294.70	thin gray pl, cub	8.0	880	d	i	...	s NH_4OH, h HNO_3
s184	stearate	$AgC_{18}H_{35}O_2$	391.35	wh powd amorph	...	205	...	0.006^{20}	...	0.006^{25} al; 0.006^{25} eth
s185	sulfate	Ag_2SO_4	311.80	wh, rhomb, 1.7583, 1.7748, 1.7852	$5.45^{29.2}$	652	d 1085	0.57^{0}	1.41^{100}	s a, NH_4OH; i al
s186	sulfide	Nat. acanthite, Ag_2S	247.80	gray-blk, rhomb	7.326	tr 175	d		v sl s	s KCN, conc H_2SO_4, HNO_3
s187	sulfide	Nat. argentite, Ag_2S	247.80	blk, cub	7.317	825	d	8.4×10^{-15}		s KCN, a
s188	sulfite	Ag_2SO_3	295.80	wh cr	...	d 100	...	v sl s	...	s a, NH_4OH, KCN; i HNO_3
s189	*d*-tartrate	$Ag_2C_4H_4O_6$	363.81	wh, scales	3.423^{15}	d	...	0.2^{18}	0.203^{25}	s a, KCN, NH_4OH
s190	*ortho*tellurate, tetra-H	$Ag_2H_4TeO_6$	443.40	straw yel, rhomb bipyr	...	d>200	...	i	i	s KCN, NH_4OH
s191	telluride	Nat. hessite, Ag_2Te	343.34	gray, cub	8.5	955	...	i	i	s KCN, NH_4OH
s192	tellurite	Ag_2TeO_3	391.36	yel-wh ppt	...	250-bl 450-pa yel	...	i	i	s KCN, NH_3
s193[1]	thioantimonite	Nat. pyrargyrite, Ag_3SbS_3	541.55	red, trig, 3.084 2.881 (Li)	5.76	486	...	i	i	s HNO_3
s193[2]	thioarsenite	Nat. proustite, Ag_3AsS_3	494.72	scarlet red, trig, 3.088, 2.792	5.49	490	...	i	i	s HNO_3
s194	thiocyanate	AgSCN	165.95	col cr	...	d	...	0.000021^{25}	0.00064^{100}	s NH_4OH; i a
s195	*di*-thionate	$Ag_2S_2O_6.2H_2O$	411.90	rhomb cr, ~1.662	3.61	...	...	...	...	...
s196	thiosulfate	$Ag_2S_2O_3$	327.87	wh cr	...	d	...	sl s	...	s $Na_2S_2O_3$, NH_4OH
s197	tungatate	Ag_2WO_4	463.59	pa yel cr	...	...	...	0.05^{15}	...	s KCN, NH_4OH, HNO_3
	Silver complex									
s198	diamminesilver *per*-rhenate	$[Ag(NH_3)_2]ReO_4$	392.13	col monocl cr	3.901	...	...	...	...	1.618 conc NH_4OH
s199	**Sodium**	Na	22.9898	silv, met cub, 4.22	0:97	97.81 ± 0.03	882.9	d to NaOH + H_2	...	d al; i eth, bz
s200	acetate	$NaC_2H_3O_2$	82.03	wh gr powd, monocl, 1.464	1.528	324	...	119^{0}	170.15^{100}	sl s al
s201	acetate trihydrate	$NaC_2H_3O_2.3H_2O$	136.08	col, monocl pr, effl, β 1.464	1.45	58	123, $-3H_2O$, 120	76.2^{0}	138.8^{50}	2.1^{15} al; s eth
s202	alumina trisilicate	Nat. albite, $NaAlSi_3O_8$ (or $Na_2O.Al_2O_3.6SiO_2$)	262.22	col, tricl, 1.525, 1.529, 1.536	2.61	1100	...	...	sl d	s HCl; d dil al
s203	*meta*aluminate	$NaAlO_2$	81.97	wh amorph powd, hygr, 1.566, 1.595, 1.580	...	1800	...	s	v s	i al
s204	aluminum chloride	$NaCl.AlCl_3$	191.78	wh-yelsh cr powd, hygr	...	185	...	s	s	...
s205	aluminum *meta*- ...	Nat. jadeite.	404.28	col, monocl	3.3	1000–1060	...	i	i	d HCl